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ELECTROPLATED NICKEL RAIN EROSION RESISTANT COATING

JAMES H. WEAVER

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FOREWORD

This report was prepared by the Elastomers and Coatings Branch, Nonmetallic Materials Division, Air Force Materials Laboratory, Air Force Systems Command, Wright-Patterson Air Force Base, Ohio. The work was initiated under Project No. 7340, "Nonmetallic and Composite Materials," Task No. 734007, "Coatings for Energy Utilization, Control and Protective Functions," and was administered under the direction of the Air Force Materials Laboratory, Mr. James H. Weaver (MANE), Project Engineer.

This report covers work conducted from July 1965 to September 1966. The report was submitted by the author in August 1967.

The author gratefully acknowledges the assistance of Mr. Roger Vissoc, University of Dayton Research Institute, during the experimental portion of the program.

This technical report has been reviewed and is approved.

W. P. Johnson

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ABSTRACT

The problem of protection of plastic components of advanced aircraft and missile weapon systems from the damaging effect of rain impingement at high speeds is severe and will become more severe in the future. Metal coating of plastic laminates is particularly applicable to plastic structural members such as wing leading edge of aircraft, helicopter rotor blades, and turbine engine compressor blades.

Epoxy, polyester, and polybenzimidazole laminates were electroplated with a minimum of 12 mil of nickel and exposed to a rain environment at subsonic speeds. The plated epoxy endured 160 minutes, the plated polyester 180 minutes, and the plated polybenzimidazole 473 minutes with no visible evidence of erosion. The latter represents a 40-fold increase in resistance over specification neoprene coatings. Supersonic rocket sled test runs at Mach 1.5 and 2.0 showed no evidence of erosion. The 60-degree angle exposure at Mach 2.5 showed mild damage. Electroformed nickel patches were bonded to stabilizer edges of an F-100 airplane which subsequently made a total of 163 penetrations into rain and hail storms for a total time of 440 minutes with no indication of erosion of the electroformed nickel.

Substrate preparation and nickel thickness were found to be the most important criteria for obtaining good rain erosion resistant coatings. Mechanical interlocking achieved by sandblasting appears to be the most efficient method to obtain adhesion to the inert surface. A minimum thickness of 12 mil of the nickel coating on the laminates is recommended to obtain protection from rain erosion.

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SECTION I

INTRODUCTION

The phenomenon of rain erosion has been described by Wahl (Reference 1). During the research effort covered by this report, however, the effect of surface roughness on the erosion resistance of nickel electroplated laminates was noted. The suggested mechanism is that a rough surface breaks the raindrop into small fragments and consequently has less severe impingement impact. The smaller particles also produce less radial flow and the shear stresses are smaller. This theory has been studied and is described in this report.

SECTION II

NICKEL COATING PROPERTIES

Nickel is the most widely used metal for engineering applications because of the mechanical properties obtainable and a more profound knowledge of nickel-plating solutions pertinent to this type of application. By proper selection of a nickel bath and its operating conditions, one can control hardness, density, tensile strength, stress, and rate of deposition to meet almost any design requirement. Excessively high stresses resulting from certain nickel baths can cause peeling, cracking, crazing, warping, blistering, distortion, shrinkage, and even complete destruction and failure of structural units or protective coatings. Howeve a nickel sulfamate bath is capable of producing the type of mechanical properties desired for rain erosion protection. The properties considered most important are high hardness, increased ductility, and low tensile stress. Typical mechanical properties produced by sulfamate nickel baths are:

Hardness	150 to 350 VHN
Tensile Strength	60,000 to 100,000 psi
Elongation in 2 in.	10 to 30%
Internal Stress	500 to 7000 psi, tensile

Several investigators have correla the internal stress levels with processing conditions (Reference 2). Where higher current densities are required to achieve desired mechanical properties, stress reducers can be used to maintain the desired stress values. These stress reducers will also increase hardness which is desirable for rain erosion protection.

SECTION III

EVALUATION TECHNIQUES

A whirling arm facility located at Wright-Patterson Air Force Base was used for the subsonic evaluations. This facility included a 6 ft diameter steel blade mounted on a 100-hp motor. The rain system was composed of hypodermic needles selected for proper drop size (1.5 to 2.0 mm diameter) on a 6-ft diameter pipe ring. A stroboscopic unit and periscope arrangement enabled observation of the specimen while running (see Figures 1 and 2). The velocity was 500 mph and 2.0 inches per hour of rainfall.

For supersonic evaluations a rocket sled facility was used. Multiple flat samples were placed on a wedge in positions such that impact with the rain-drops would be at various angles. The wedge was mounted on a sled which was propelled by rocket motors through a calibrated rainfall at the desired velocities. The sled test facility is located at Holloman Air Force Base, New Mexico, and operates on a monorail 35,000 ft long. The facility has a 6000 ft long artificial rainfall. The test velocities ranged from Mach 1.5 to 2.5 and the rainfield produced 2.5 inches per hour. The water droplet sizes range from 1 to 2 mm in diameter. Figure 3 shows the wedge mounted on the front of the rocket engine.

SECTION IV

EXPERIMENTAL

1. SUBSTRATE MATERIAL

Polybenzimidazole (PBI) was chosen as representative of high temperature, high strength plastic materials being considered for future applications such as helicopter blades, leading edges, and vertical stabilizers. Reinforced epoxies and polyesters were chosen as typical representatives of materials used for present day applications.

2. SUBSTRATE PREPARATION

The preparatory treatments of the inert plastic surface govern the success or failure of any process for application of the initial chemically reduced film. The same processing procedures were used for all plastic substrate materials.

The formation of a suitable conductive and adherent metallic film on a plastic surface involves the following steps:

1. Roughening or deglazing
2. Cleaning the surface
3. Conditioning the surface
4. Sensitizing the surface
5. Activating the surface
6. Forming the chemical film by chemical reduction

The above processing steps are described in detail in Reference 3.

3. SPECIMEN CONFIGURATION

During this research effort, the sample configuration for the whirling arm evaluation rig varied. Figure 4 shows the two configurations used. The current density distribution varied from the edge of the specimen to the center of the curvature. Since the raindrop impingement is on the curved portion of the specimen, thickness determinations were made at this point. Thickness was determined by sectioning a sample at the leading edge, and mounting, polishing and measuring the plating thickness on photomicrographs. Figure 5 shows the thickness of three samples. Each unit is equal to 1 mil. The samples for the sled tests were flat and 1.25 x 1.25 x 0.25 inches in size.

SECTION V

RESULTS

1. ADHESION

Substrates of epoxy were roughened by the various conditions outlined in Reference 3. They were sensitized, activated, and plated for 10 minutes in the electroless copper bath. Some of the light and heavy sandblasted samples were given a copper flash and were nickel-plated for five minutes to check the adhesion of the combined plating system. The samples were crossmarked with a sharp instrument using as much pressure as possible. One inch Scotch tape was applied immediately to the crossmark. The tape was pressed tightly against the surface of the coating using the thumbnail to work the tape into the rough surface. The tape was removed with one quick 180-degree motion. The results of the tape test are shown in Figure 6. It can be seen that the sandblasted samples gave superior adhesion as compared to those prepared using the acid conditioner. The adhesion of the combined plating system was very good. The proprietary conditioner was used to condition the samples and the results are shown in Figure 7. The light sandblasting and the proprietary conditioner gave excellent adhesion.

The adhesion of the electroless copper to the substrate had been determined by qualitative tests (tape test), but it was deemed important to have a quantitative measure of the strength of the bond between the electroless copper and the substrate. The test used consisted of bonding the electroless copper to an epoxy substrate with an adhesive, and then measuring the force needed to pull the assembly apart. Figure 8 shows the assemblies after failure. Numbers 2 and 3 failed in the adhesive and epoxy laminate, respectively, while number 1 was a cohesive failure in the copper. Failure occurred at 450 psi. In all cases the failure occurred in the assembly prior to failure of the bond between the electroless copper and the substrate. Mechanical interlocking such as achieved by the sandblasting appears to be the most efficient method to obtain adhesion of the electroless copper to the inert surface.

2. EFFECTS OF THERMAL CYCLING

Since the leading edge of a high speed component would be subjected to thermal cycling during operation, a thermal cycling evaluation was initiated. Two samples of each substrate material which had been electroplated with nickel (12 to 16 mil) were thermal cycled. The maximum temperature exposure for the epoxy was 149°C (300°F), the polyester 122°C (250°F), and the PBI 313°C (600°F). These samples were heated at these temperatures for 30 minutes, air cooled, and the cycle repeated 10 times. There was indication of blistering on one polyester sample. All other samples, however, were free of visual defects. These samples were exposed to the simulated rainfall at 500 mph an additional 50 minutes to determine if any damage had been caused by the thermal cycling. It was determined that the thermal cycling produced no adverse effects on the rain erosion resistance of the nickel-plating.

3. EFFECTS OF NICKEL-COATING THICKNESS

The thickness of the nickel deposit is very critical for successful rain erosion resistance. In the initial stages of the research, thicknesses in the range of 4 to 8 mil were evaluated. The times-to-failure were greatly improved over the neoprene coatings (rain erosion specification material) when exposed to the 500 mph in 2-inch per hour simulated rainfall. Neoprene coatings 10 to 12 mil thick failed in 5 to 8 minutes when exposed to the rain environment at 500 mph. Increased thickness of the nickel seemed feasible so thicknesses were then increased up to 16 mil. Table I shows the results obtained.

TABLE I

EFFECT OF THICKNESS ON FAILURE OF NICKEL-PLATED LAMINATES

Thickness (mil)	Time-to-Failure (min) Polyester	Time-to-Failure (min) Epoxy	Time-to-Failure (min) PBI
4	5		12
6	6	10	68
8	42	51	106
10	138	88	
12	180	160	473
14	364	264	
16			840

The increased resistance to rain erosion for the PBI laminates may be due to the smaller radius leading edge. The thickness of coating on the smaller radius leading edge may be greater than on the larger radius. A correlation between thickness and plating variables was based on the larger radius leading edge. Increased current density could have resulted on the smaller leading edge under the same process conditions, thus causing increased coating thickness.

Figure 9 shows the "as-received" PBI laminate and after "sandblasting." The sandblasting removed the glaze and roughened the surface to improve the adhesion. Figure 10 shows an uncoated PBI laminate after 6 minutes exposure to the simulated rainfall at 500 mph. Erosion is very severe and would erode through the laminate and cause failure of the component. Figure 11 shows the results after 46 minutes exposure of a deposit 6 mil thick. This is a significant improvement over the uncoated laminate. Figure 12 shows a 16-mil nickel-plated PBI laminate after 840 minutes exposure without failure. The water impingement has only polished the nickel surface. Figure 13 shows the results obtained on the PBI, epoxy, and polyester laminates for time vs thickness. In all cases the [illegible] of the curves is the same with the coated PBI laminate showing superior resistance over [illegible] epoxy and polyester.

4. EFFECTS OF SURFACE ROUGHNESS

The effects of surface roughness have been observed throughout this research effort. Samples were given a thicker copper flash and polished to give a smooth surface. Others were purposely given a heavier sandblast to produce a very rough surface. These surfaces were plated to an equal thickness of nickel and tested in the rain facility. Figure 14 presents the results obtained with four thicknesses of nickel.

The author believes that the rough surface breaks the raindrop into smaller particles and, consequently, has less impingement impact. These smaller particles produce less radial flow, and shear stresses are smaller. This, in turn, produces an erosion coating which is superior to the smooth surface. The PBI laminate plated to a thickness of 16 mil is very rough and did not fail after 840 minutes.

5. SUPERSONIC SLED TESTS

Subsonic evaluations of nickel electroplated laminates have indicated 40 times the resistance over the elastomeric and ceramic coatings. The ductility of the nickel (elongation up to 20%) is felt to be chiefly responsible for this resistance. Epoxy laminates (1.25 x 1.25 x 0.25 inches) were electoplated to a thickness of 10 mil and placed in the wedge at various angles (15°, 30°, 45°, 60°, and 90°). Figure 15 shows the results of the sled test at Mach 1.5, 2.0, and 2.5. At Mach 1.5 and 2.0 the nickel electroplating appeared unexposed after the firings. At Mach 2.5, specimens at all positions except 60 degrees also appeared unaffected. At 60 degrees the surface of the nickel had been deformed and ridges appeared where it had been pushed up or "wrinkled" by the radially flowing water. The 60-dgree nickel electroplated samples had lost adhesion but were not penetrated by the water drops. Future tests will include different thicknesses of nickel electroplated on other laminates and tested up to Mach 5.0.

6. PROJECT ROUGH RIDER TESTS

A practical evaluation of rain erosion resistant coatings was initiated known as Project Rough Rider. For this program, "patches" of experimental coatings were applied to various areas of an F-100 airplane which penetrated rain storms to check the erosion of materials due to repeated impact of raindrops. Electroformed nickel patches were bonded to the leading edges of each horizontal stabilizer and on the vertical stabilizer. Figure 16 shows the nickel area on the vertical stabilizer. One hundred sixty-three penetrations were made at 275 knots indicated air speed from 25,000 to 35,000 feet. The total accumulated time in the storms was 440 minutes. All electroformed nickel patches remained in excellent condition with no indication of erosion.

SECTION VI

CONCLUSIONS

Procedures have been established for nickel-plating nonconductive substrates for rain erosion resistance. By careful control, a fine grained, low stress nickel deposit which has excellent adhesion, hardness, and ductility is produced. The advanced process has been successfully applied to epoxy, polyester, and PBI laminates and extended the life of normal leading edge materials some 40 times when compared to the specification neoprene material for subsonic applications. The supersonic rocket sled test runs at Mach 1.5, 2.0, and 2.5 were very successful with only the 60-degree angle showing any evidence of damage from the raindrop impingement. The electroformed nickel patches on the F-100 airplane endured a total of 163 penetrations for a total time of 440 minutes with no indication of erosion. Superior erosion resistance is obtained by roughening the surface slightly. The same procedures were used for each substrate with equal success. It was determined that the thickness of nickel be a minimum of 12 mil on the laminates to obtain extended life of a leading edge.

REFERENCES

1. N. F. Wahl, Investigation of the Phenomena of Rain Erosion at Subsonic and Supersonic Speeds, AFML-TR-65-330, Air Force Materials Laboratory, Wright-Patterson Air Force Base, Ohio, October 1965.

2. D. A. Fanner and R. A. F. Hammond, Transactions, Inst. Metal Finishing, Vol 36, Part 2, 1958-1959, p 32.

3. J. H. Weaver, "Nickel Electroplated Nonconductive Materials for Protection from Rain Erosion," Plating, May 1967.

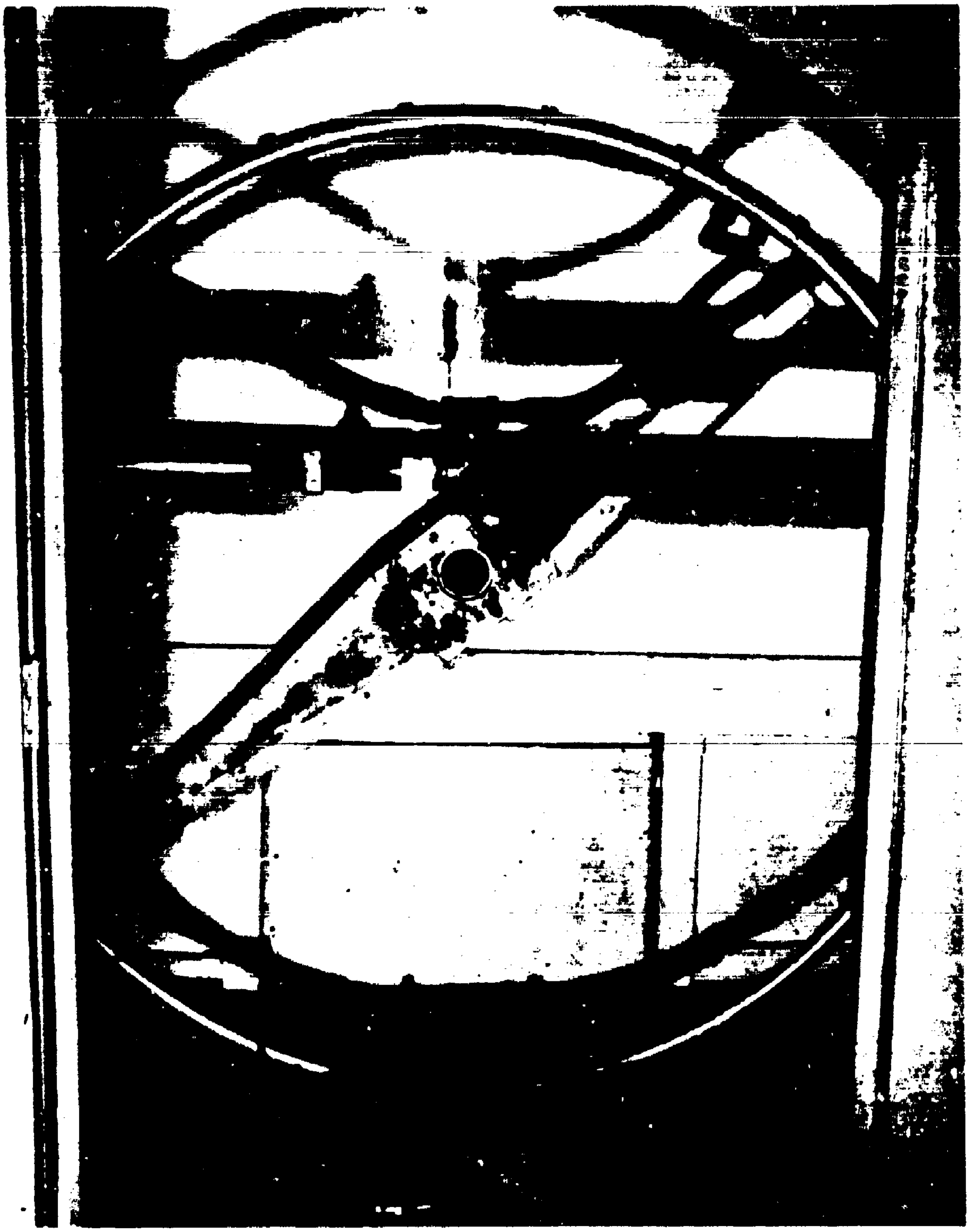


Figure 1. Rain Erosion Facility - Spray Ring and Whirling Arm

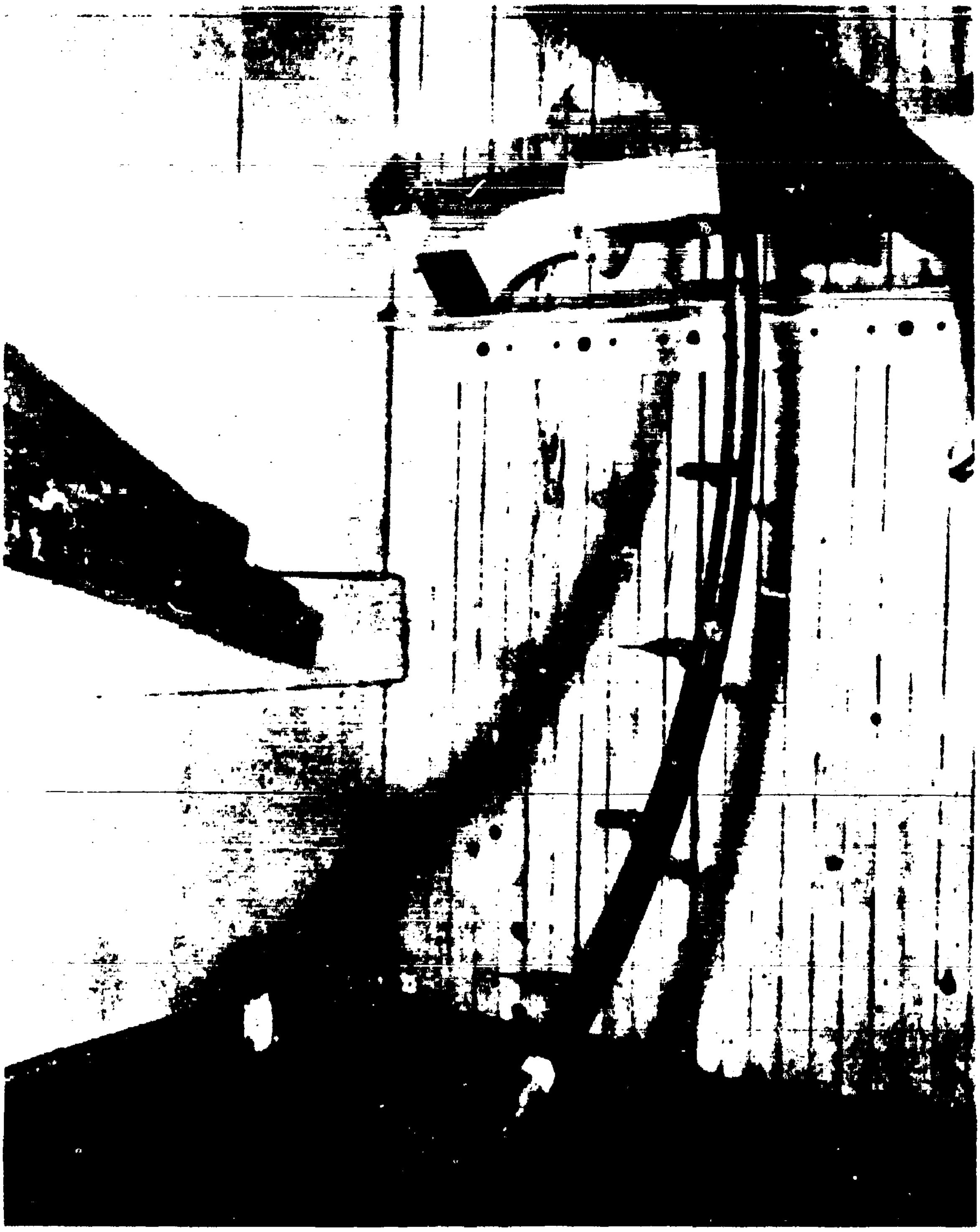


Figure 2. Rain Erosion Facility - Spray Ring, Whirling Arm With Test Specimen Installed, and Periscope Tube

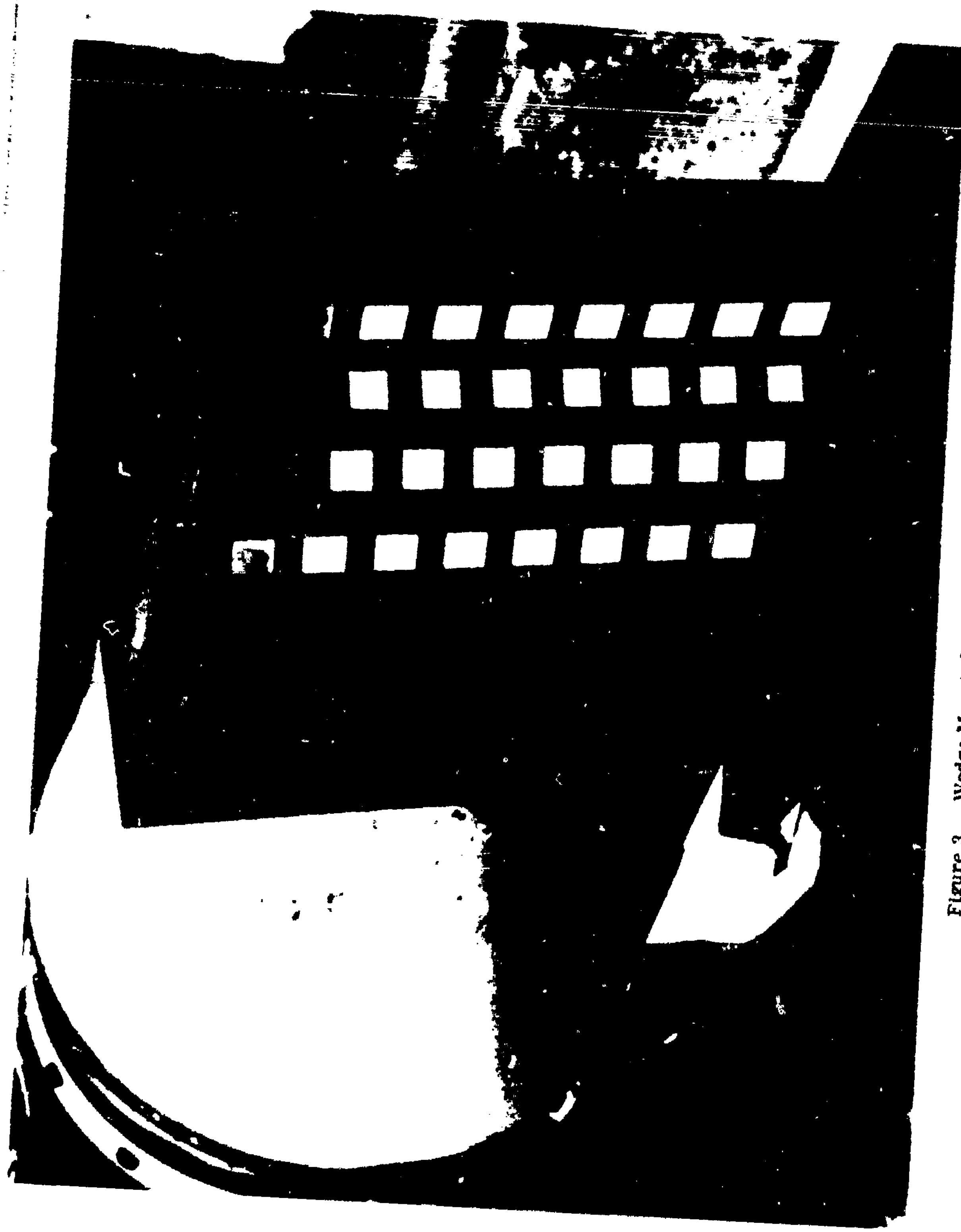


Figure 3. Wedge Mounted on the Front of Rocket Engine

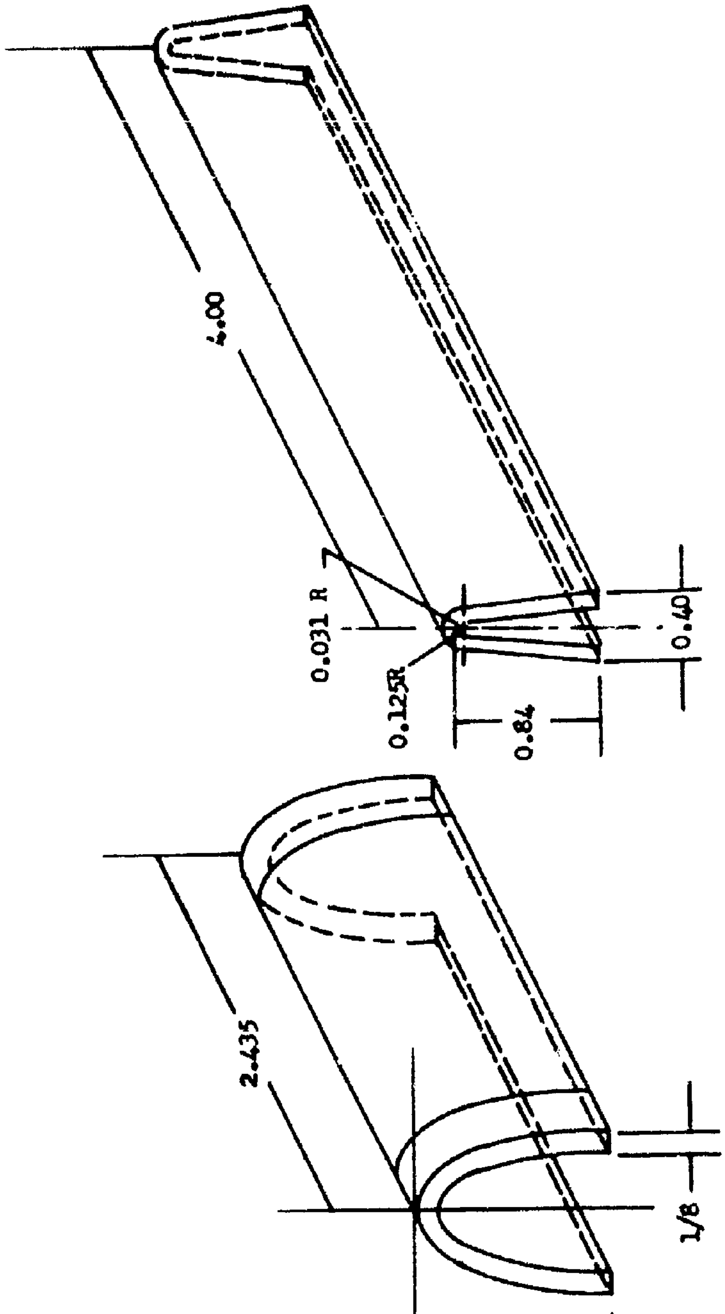


Figure 4. Specimen Configurations

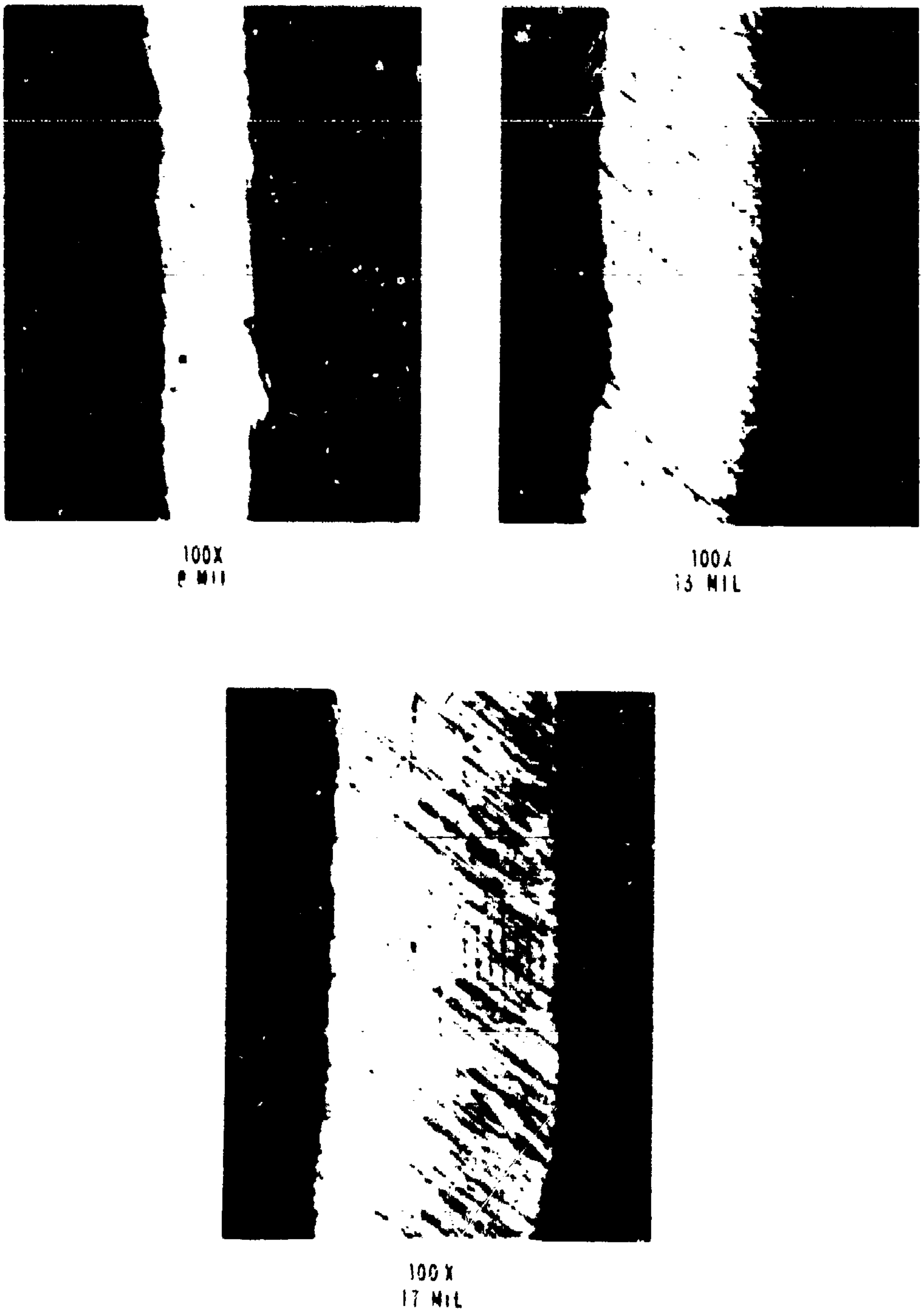


Figure 5. Photomicrograph of Three Plating Thicknesses

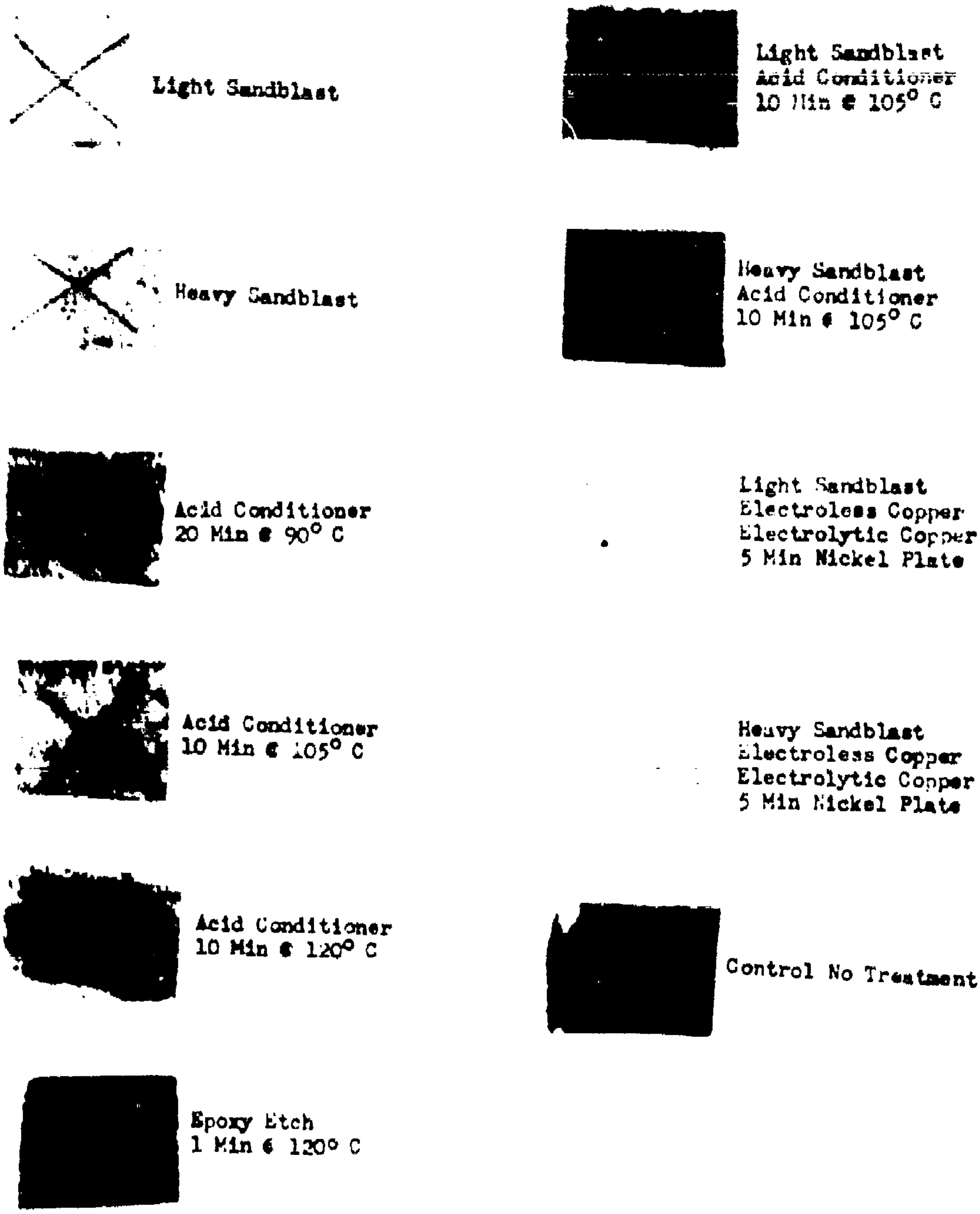
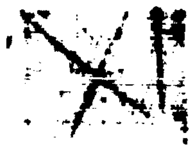


Figure 6. Adhesion Tests



Sandblast

Sandblast
5 Min Enplate Acid
Conditioner @ 60° C

Sandblast
5 Min Enplate Acid
Conditioner @ 75° C

Sandblast
5 Min Enplate Acid
Conditioner @ 85° C

Enplate Acid
Conditioner
5 Min @ 85° C

Figure 7. Enplate Conditioner Adhesion Tests

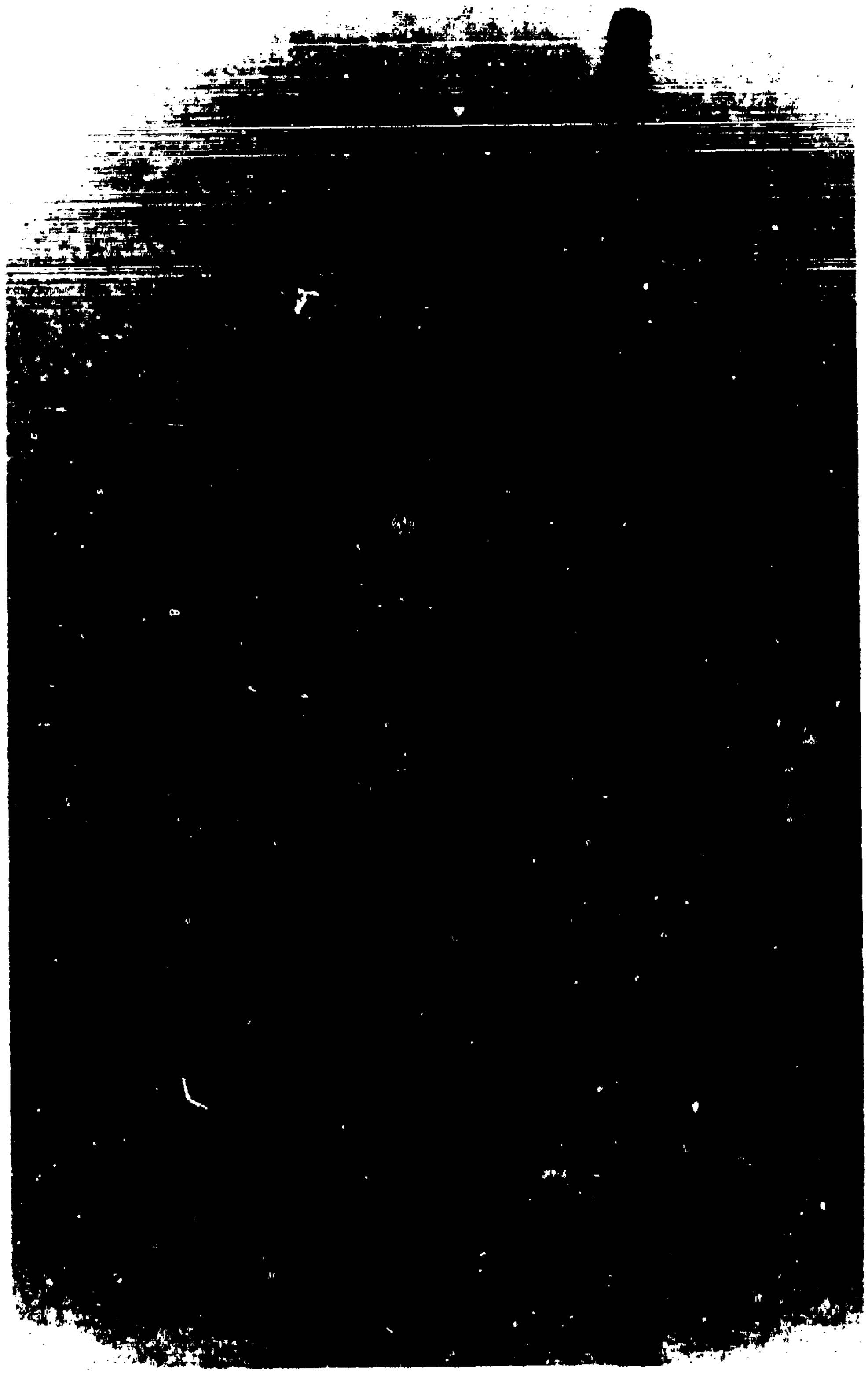


Figure 8. Assembly Adhesion Tests

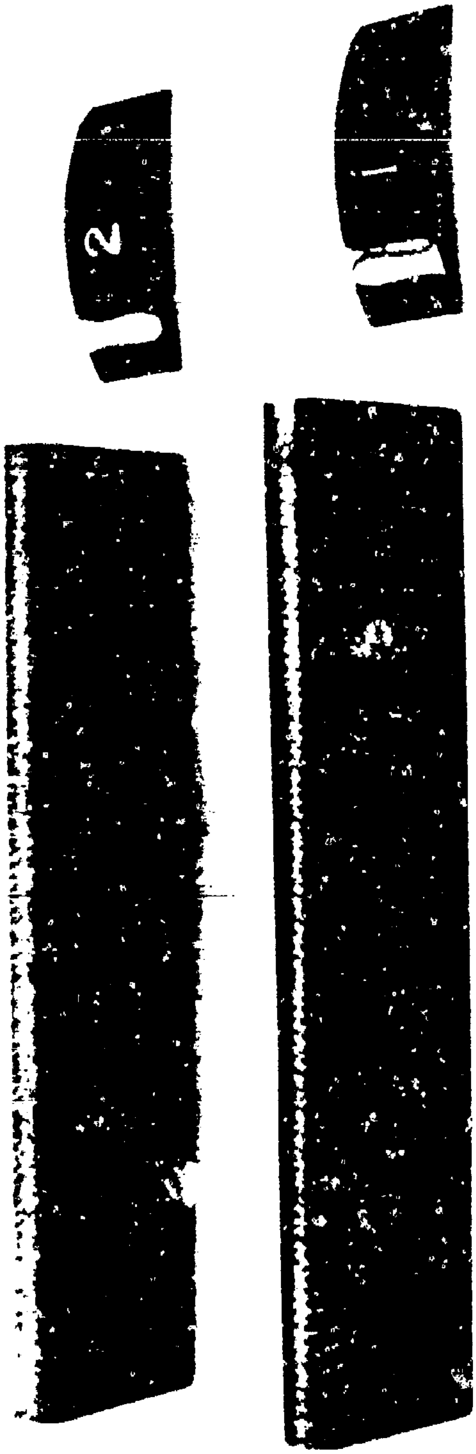


Figure 9. "As-Received" and Sandblasted PBI Specimens

Figure 10. Uncoated PBI Laminate After a Six-Minute Exposure

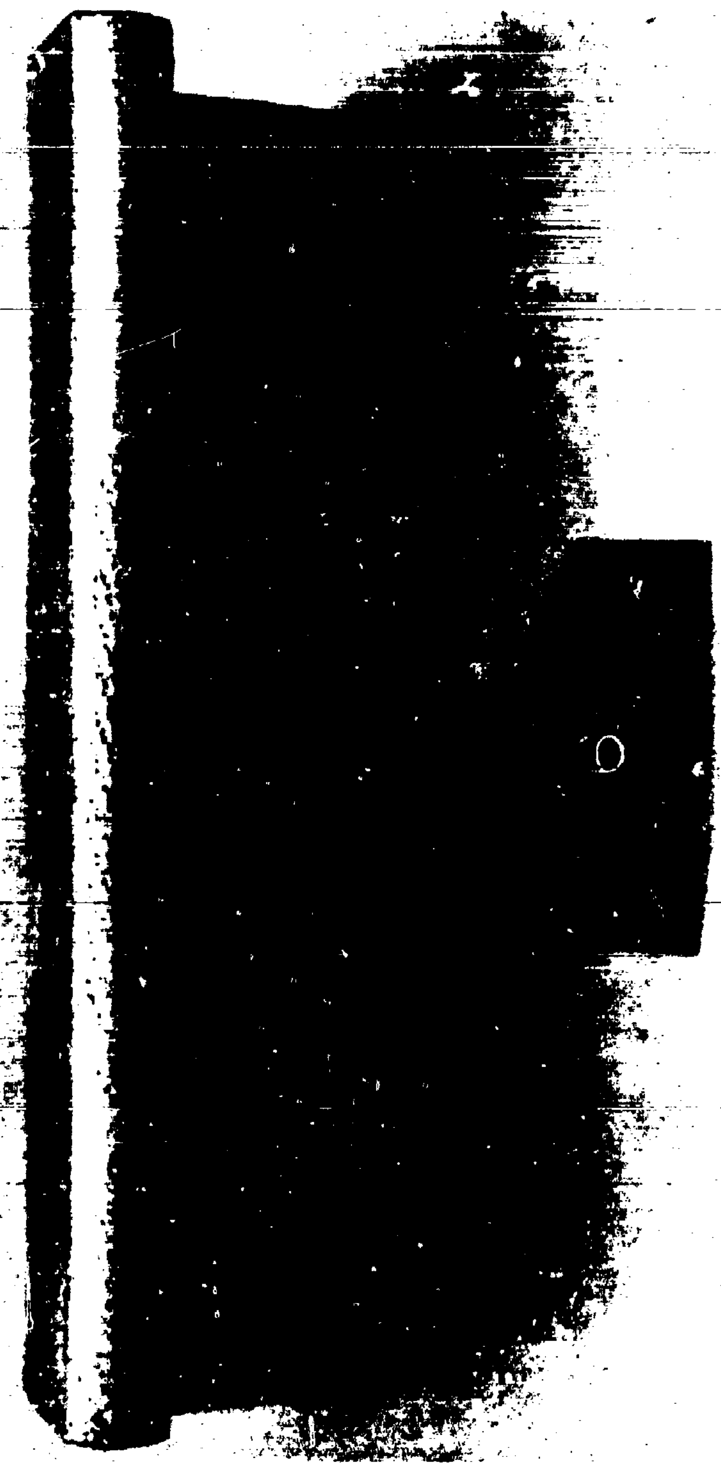


Figure 11. Six-Mil Nickel-Plated PBI After a 46-Minute Exposure

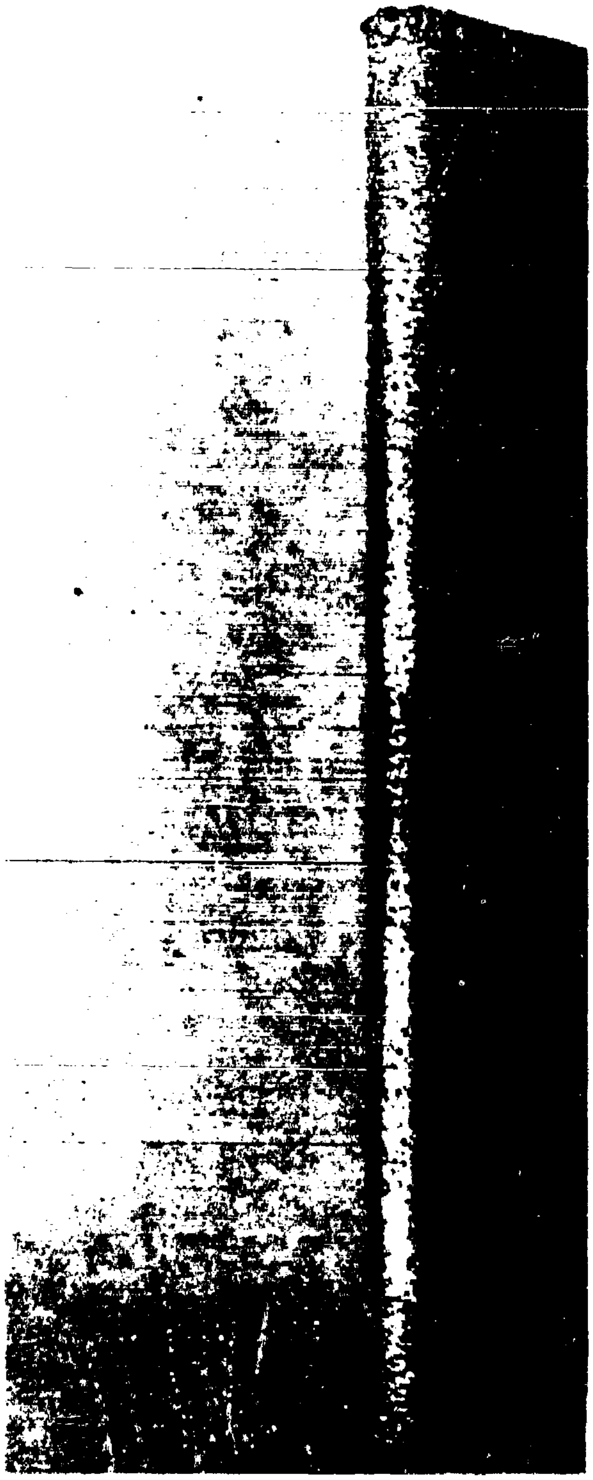


Figure 12. Sixteen-Mil Nickel-Plated PBI After an 840-Minute Exposure

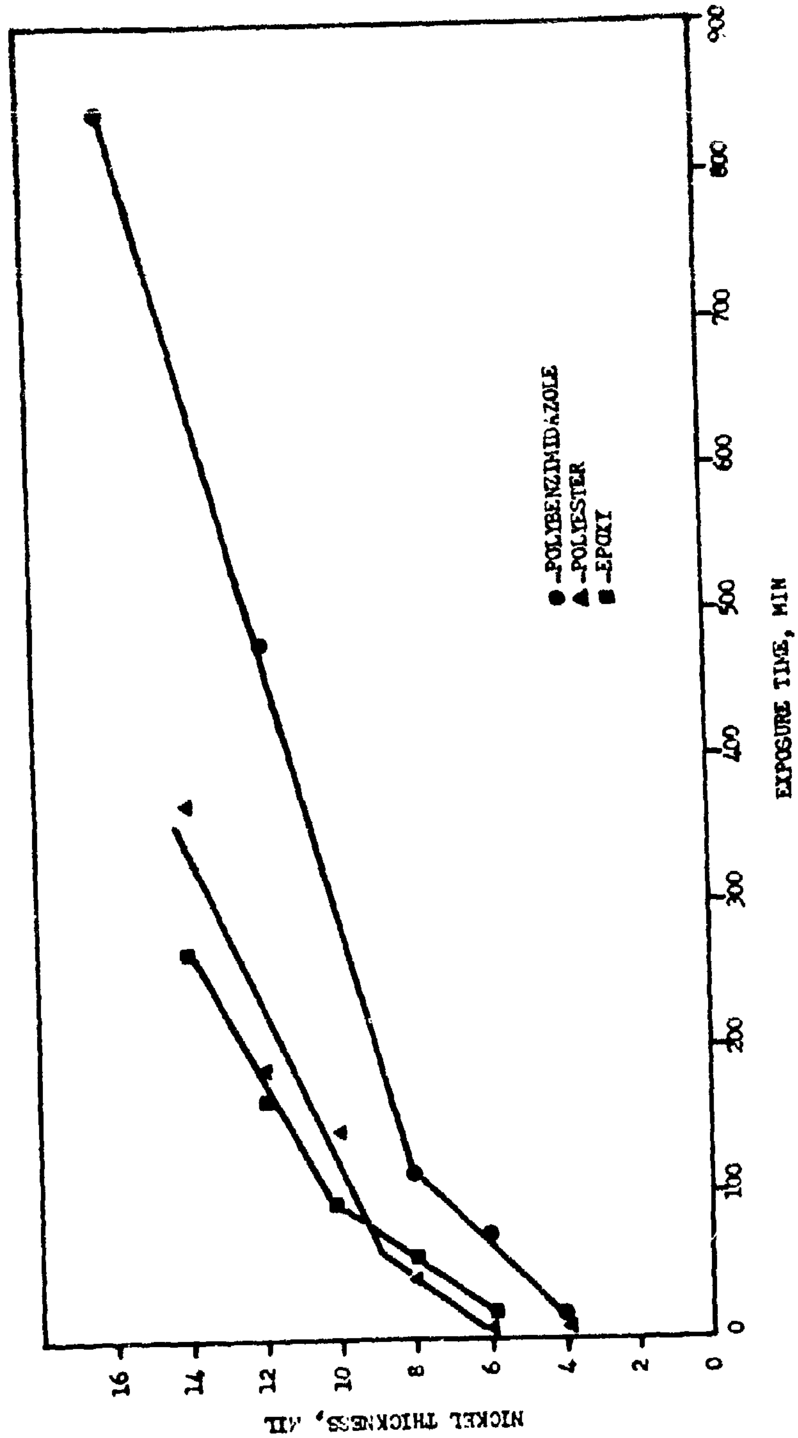


Figure 13. Thickness vs Time for Rain Erosion Coated Specimens

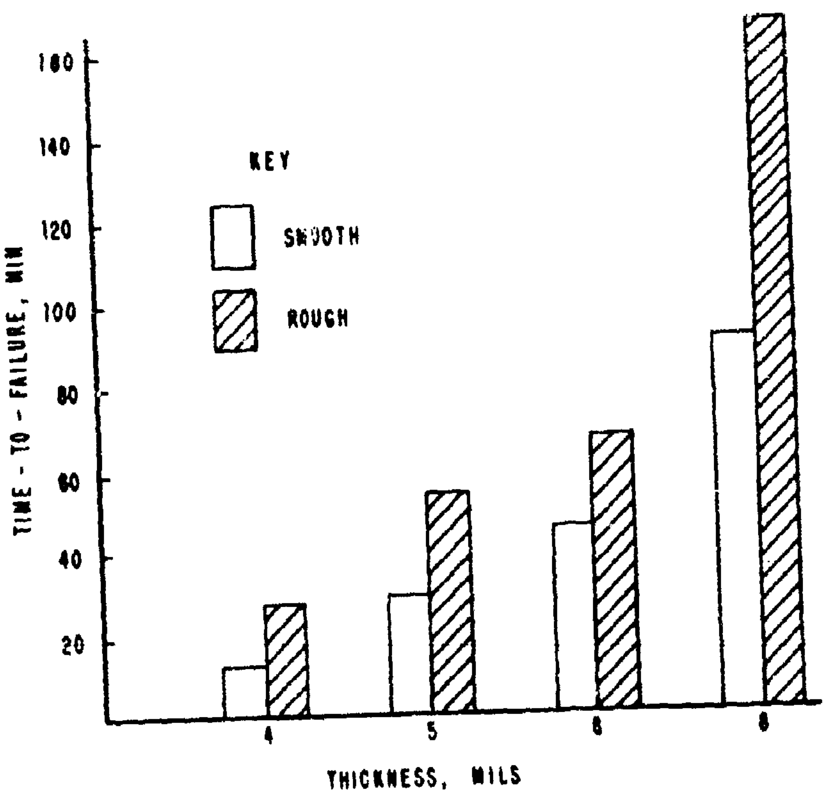


Figure 14. Effects of Smooth and Rough Surfaces on Time-to-Failure

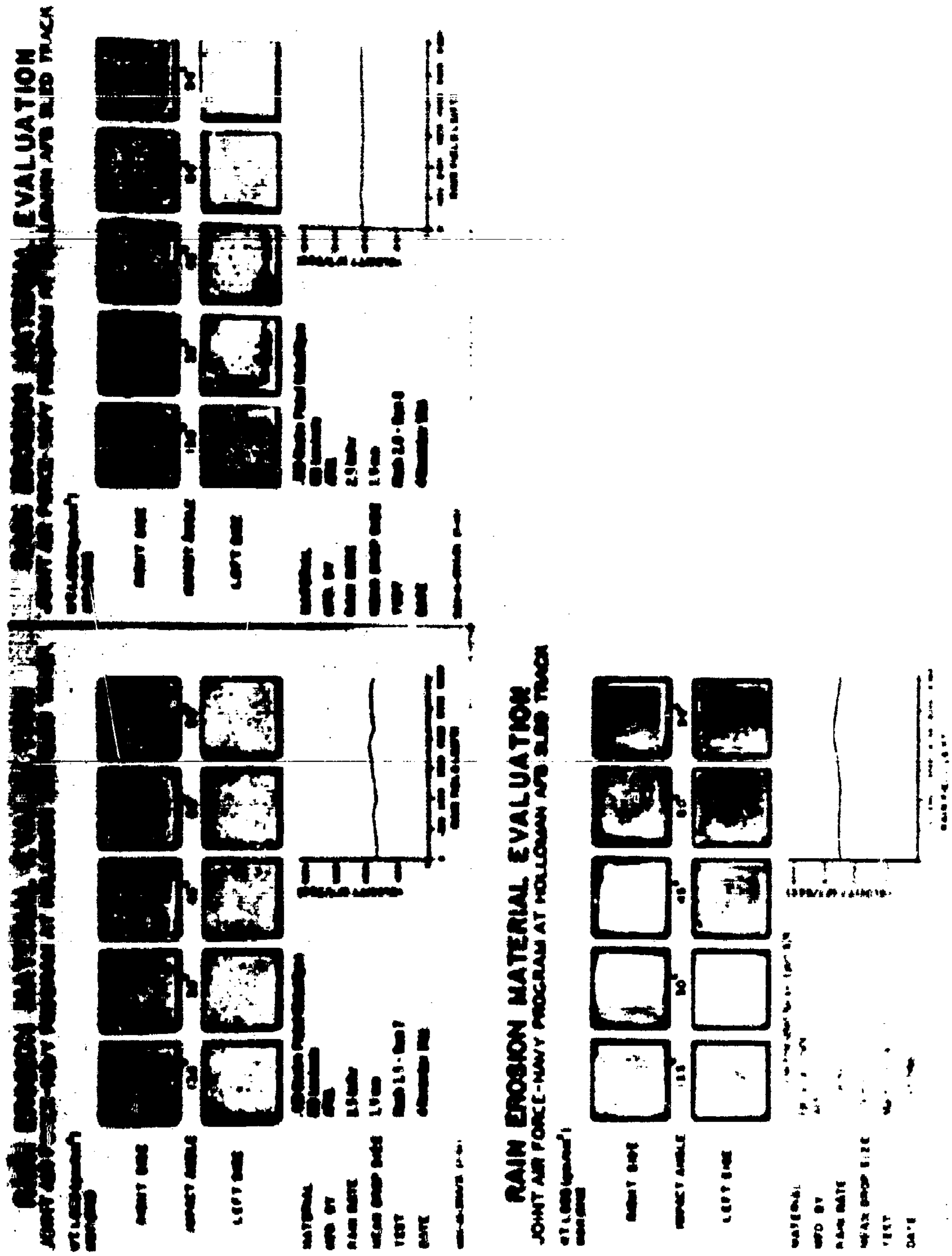


Figure 15. Sled Test Results

Figure 16. Electroformed Nickel Bonded to Vertical Stabilizer

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Rain Erosion Protection						
Nickel Electroforming						

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